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PETROGRAPHIC CHARACTERIZATION OF THICKENER FEED SAMPLES

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by

V. A. Munoz*

ABSTRACT

This report compares the petrographic characterization of three conventional thickener feed samples from the Cardinal River coal mine. It provides additional information to the existing laboratory data, and contributes to the understanding of some of the problems related to these samples during the flocculation process.

The results showed that there were diferences among the samples in relation to size of the coal particles, relative degree of degradation, reflectance values, and association with the mineral matter. The sample having the most problematic characteristics during flocculation exhibited the highest degree of degradation of the coal particles which were in intimate association with the minerals. The photomicrographs depict the severity of the morphological alteration of the coal particles, and allow the visual comparison of their size.

Surface areas of the three feeds were also determined, but no correlation between their values and the water clarification process was found.

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INTRODUCTION

Coal petrography has been applied to several technical processes such as coal getting, beneficiation, carbonization, liquefaction and combustion. In some of these applications the results of the petrographic analysis have become a technological essential information.

In the case of coal beneficiation, coal preparation engineers rely heavily on data obtained from float and sink tests and the corresponding ash determinations to calculate the washability curves. These data allow for accurate prediction of the yield of coal obtainable at a given separation density. However, the important contribution that coal petrography can make to the solution of cleaning problems is often overlooked and restricted to a few specific situations. Some of these situations are related to the determination of the possibilities of reducing the ash yield by providing information regarding the distribution and the concentration of the minerals intergrown with the coal macerals. The study of the distribution of the pyrites in tailings, its reduction in the product and its association with specific macerals can be given as examples. The analysis of misplaced material and the contribution to the finding of its causes is another use of the petrography in the coal cleaning process.

In the present study, the petrographic analysis was performed to provide information that would contribute to a better understanding of the flocculation behavior of washery thickener feed samples with respect to water clarification. At CRL the characterization of such samples for flocculation studies usually includes the determination of settling rates and related parameters including pH, total and suspended solids contents, water analysis, particle size distribution, surface charge (zeta potential), proximate and ultimate analysis. Three thickener feed samples named "August 12", "August 14", and "August 15" from the Cardinal River Coals Ltd. washery were studied to determine the factors responsible for differences in their response to flocculation. The laboratory settling tests corroborated the washery results and showed that the August 14 sample, the coarsest in particle size and the lowest in ash content (3i.4%), required the least dosage to achieve maximum clarity and gave the highest settling rate of the three feeds. On the other hand, the August 15 sample, the finest in particle size and the highest in ash content (56.7%)

required only slightly higher dosage to obtain the same clarity and was less troublesome than the August 12 sample with a larger particle size and lower ash content (37.4 %). This feed gave the most problems of clarification in the washery. The available laboratory flocculation test and analyses provide no conclusive relationships to expalain this unusual bahavior.

The petrographic analysis provided additional information related to the size of the coal particles, their morphology, the maceral composition, the association of the macerals with the mineral matter, and the relative degree of oxidation or alteration. This information on the surface condition of the coal particles is essential to achieving a complete understanding of flocculation problems and to finding practical solutions.

EXPERIMENTAL

The solids from the three slurries were extracted by evaporation of the water at 100 °C, and pulverizing the obtained cakes. It was anticipated that the large mineral matter content of the samples, as indicated by the ash contents in the range of 31 % to 57 %, could produce severe problems during the polishing of the pellets. The presence of clays, which may have a tendency to swell due to absorption of water, would make it impossible to obtain acceptable polishing for the purpose of microscopic examination. Consequently, the petrographic sample preparation of the thickener feed samples was carried out by applying a modified procedure.

The pellets were prepared by mixing 4 grams of the solids with 7 grams of epoxy resin. The large quantity of epoxy resin was used to prevent excessive swelling of the clays and the problems that would be associated with it. The grinding and polishing were done using the standardized procedure used at CRL. A Carl Zeiss research microscope-photometer system was used for the microscopic study. Because of the small particle size of the solids, the magnifications were much larger than are normally used in petrographic analysis. The combination of lenses provided a 2000 times magnification. Different magnifications were used for the photomicrographs as indicated in the corresponding figures.

Only 200 points per sample were counted for determination of the maceral composition because of the difficulties arising from the small particle size and because of the low ratio of solids to epoxy in the pellets. In the case of random reflectance, 75 measurements per sample were taken in the vitrinite component using polarized light with the monochromator set at 540 nanometers.

The surface areas were determined using the standard B.E.T. (Brunauer, Emmett, and Teller) method in an Accusorb 2100 E physical adsorption analyzer. Nitrogen was used as adsorbate at liquid nitrogen temperature.

RESULTS AND DISCUSSION

The results of the petrographic analyses of the three thickener feed samples studied are presented in Table 1. The macerals were divided into the three major groups: vitrinites, exinites, and inertinites. Further division into the maceral components was not considered necessary and in addition, it was extremely difficult to distinguish some of the macerals due to the advanced stage of degradation of many of the particles. The maceral composition, expressed as volume per cent mineral matter free basis, showed no significant differences among the samples and were all within the error of the determination (± 2%) of one another. Photomicrographs of each sample are shown in Fig. 1 to 10 where examples of the typical vitrinite and inertinite components are depicted at 1500 X; a more general view giving some indication of the size distribution is shown at 550 X magnification.

It is of interest to note that no macerals of the exinite group were found in any of the samples. Taking into account their botanical precursors (waxes, oils, sporopollenins, resins, and cutins), these components posses a more aliphatic structure and would be more hydrophobic than the vitrinites and inertinites. Therefore, it is conceivable that any particles containing exinites would have been recovered in the clean coal fraction in preference to the tailings (thickener feed) during beneficiation by flotation. On the other hand, the vitrinites were the main components of the samples. These are the macerals most susceptible to oxidation and weathering and they were found to be commonly associated with inertinites,

which are the least reactive components and the most friable. Inertinites are usually concentrated in the finer coal fractions and inportant portions are usually lost to the tailings in the flotation process.

The most important differences between the three samples were found to be in their microscopic characteristics and in the size of the coal particles. Significant but variable degrees of alteration in the morphology of the macerals is evident. The microscopic characteristics are consistent with low temperature oxidation of the coal particles and suggest that the alteration can probably be attributed to natural oxidation processes including weathering. This is indicated by the high microscopic porosity of the particles, the presence of microcracks and microfissures, particularly in the vitrinite. However, it is not possible to determine with certainty whether the oxidation took place before or as a consequence of mining, processing and/or storage in the laboratory. On the other hand, it could be conjectured that such material would have ended up in the flotation tailings (thickener feed) very likely because of the poor flotability resulting from oxidation that occurred some time before processing. It is possible that the samples were oxidized prior to processing. The oxidized macerals are more friable and more susceptible to further oxidation as comminution takes place. However, the morphology of the three samples suggests that most of the oxidation occurred during processing and/or storage in the laboratory, and could overshadow the characteristics of naturally oxidized coals.

The microscopic differences observed between the three samples, morphology, reflectance, and size of the coal particles suggest parallel changes and differences in their chemistry. Nevertheless, the high concentration of mineral matter, particularly clays, can have masking effects during the determination of some chemical differences. For example, the clays can have buffering action during pH measurements of the slurries. Because of their large suface area, usually in the range of 20 to 30 square meters per gram, clays can prevent the detection of smaller differences in surface area between the coal particles. This masking effect of the clays can interfere with any analytical technique which measures bulk properties. Microscopic analysis is not affected, however, because it determines the characteristics of individual coal particles and the bulk properties are obtained statistically. Clays can also lead to degradation of coal

particles during the laboratory storage since it is known that they catalyze chemical reactions in the coal structure given certain conditions.

The photomicrographs of Fig. 1, 2, 3, and 4 depict particles of the August 12 sample. The highly porous surface of the particles (Fig. 1 and 2) and the large number of microholes indicates a high degree of degradation. The centre of Fig. 4 shows a coal particle that has completely disintegrated with the subparticles remaining together, in intimate association with the clays. These features were common in this sample whereas they were not found in the other two. The microscopic study demonstrated that the August 14 sample consisted of the least degraded material. The surface of the particles was more homogeneous, had less microscopic porosity and contained smaller number of holes as can be observed in the photomicrographs of Fig. 5, 6, and 7. With respect to the size of the coal particles the August 14 sample presented the largest particle size while the August 15 sample showed the finest. This is evident in the photomicrographs of Figs. 3, 7, and 8 which depict the general microscopic aspect of the three samples at 550 times magnification. The photomicrographs of Figs. 8, 9, and 10 show particles of the August 15 sample. Only a small number of coal particles of the size exhibited in Fig. 9 were found. The microscopic characteristics of this sample indicate that although the particles were finer, there was less degradation compared to the August 12 sample. The relative differences in particle size are in agreement with a previous size analysis performed with the Coulter Counter. However, it is necessary to emphasize that size comparisons from petrographic analyses relate mainly to the coal particles whereas the data obtained by the Coulter Counter technique reflect the size of all the particles in the samples including the mineral components.

Reflectance of the vitrinite is not a sensitive parameter to determine oxidation. However, at more advanced stages of oxidation produced by low temperature reactions, the reflectance values decrease. In the case of the thickener feed samples studied, the mean random reflectance values corroborate the conclusions based on the morphology of the samples. The August 12 feed had the lowest reflectance value (0.99 %) which corresponds to its higher stage of degradation. The reflectance of some of the particles could not be measured because of their extremely porous surfaces caused by the degradation. This applied in particular to the

particles in the August 12 and August 15 samples. The August 14 sample gave the highest mean reflectance value, indicative of its lover relative degradation. The corresponding reflectograms are presented in Figs. 11, 12, and 13 and are plotted at the same scale. The August 12 sample exhibited the largest standard deviation of the distribution of reflectance values (0.148). This could indicate differences in susceptibility of the particles to degradation producing a wider range of reflectance values in this sample. The reflectograms of August 14, and August 15 samples shov a discontinuity which could also be attributed to differences in the degradation susceptibilities among the particles; more altered particles exhibited lower reflectance values. The mean reflectance value of the vitrinite is the parameter used in coal petrography to determine the rank of coal samples. In this case the rank assignment of these samples could not be done due to the advanced degree of degradation of the vitrinite component.

It is necessary to take into consideration the possible effects of pulverization on the coal particle size observed during the microscopic examination, and in the photomicrographs. The size and shape of the particles could have been altered during this operation and may not be the same as in the original samples. In order to circumvent this possibility, the slurries should preferably be dried in a manner to prevent the formation of cakes.

The surface area of solids depend on the particle size distribution, consequently, its determination is carried out on stage crushed samples to minimize the effects of differences in particle size among the samples. The standard particle size is usually in the range of 0.25 to 0.60 mm diameter. In the case of the thickener feed samples, the surface areas were determined on the pulverized solids and reflect the size differences of the coal and mineral components among the samples (Table 1). The August 15 feed with the highest ash content (56.7%) resulted in the largest surface area, 6.17 m^2/g, influenced mainly by its mineral components. The August 12 and August 14 samples had surface areas of 2.59 m^2/g and 1.66 m^2/g respectively. These results suggest that, in this particular situation, the surface areas of the feeds did not have significant influence in the water clarification processes. In accordance with the surface area values the treatment of the August 15 feed should have

been the most troublesome, and this was not the case. Therefore, other parameters such as the surface chemistry of the coal and mineral components played the most important role in the water clarification processes.

CONCLUSIONS

1. The August 12 sample contained the most degraded coal particles and the August 14 sample the least altered.
2. The August 15 sample exhibited the smallest coal particle size whereas August 14 sample has the largest particle size.
3. The only maceral association was vitrinites-inertinites and no vitrinites-exinites combination was found.
4. There were no significant differences in the maceral composition of the three samples.
5. The August 12 sample presented the lowest mean random reflectance and the largest standard deviation of the reflectance values distribution as a result of its high degree of degradation.
6. It is not possible to determine if the samples were oxidized before processing and a study of the fines of the raw coal would be necessary to assess this situation.
7. The surface areas reflected the size differences of the coal and mineral components of the samples. The August 15 feed had the largest surface area, 6.17 m^2/g, the August 14 resulted with 1.66 m^2/g and the August 12 with 2.59 m^2/g. These results corroborated the results of the petrographic, and Coulter Counter analyses with respect to particle size.
8. No correlation between the surface areas of the feeds and the difficulties during the water clarification processes were found.

ACKNOWLEDGMENTS

The author wishes to express his appreciation to J. L. Picard for correcting and editing this report, and to R. Frenette for the information regarding the samples studied.

Table 1 - Maceral composition, volume %, mineral matter free basis, and mean random reflectance of Cardinal River conventional thickener feed samples

PARAMETER	SAMPLE		
	August 12 1985	August 14 1985	August 15 1985
Vitrinite	67	68	66
Exinite	0	0	0
Inertinite	33	32	34
Mean Random Reflectance	0.99	1.12	1.04
Standard Deviation of Reflectance	0.148	0.099	0.136
Surface Area m^2/g	2.59	1.66	6.17

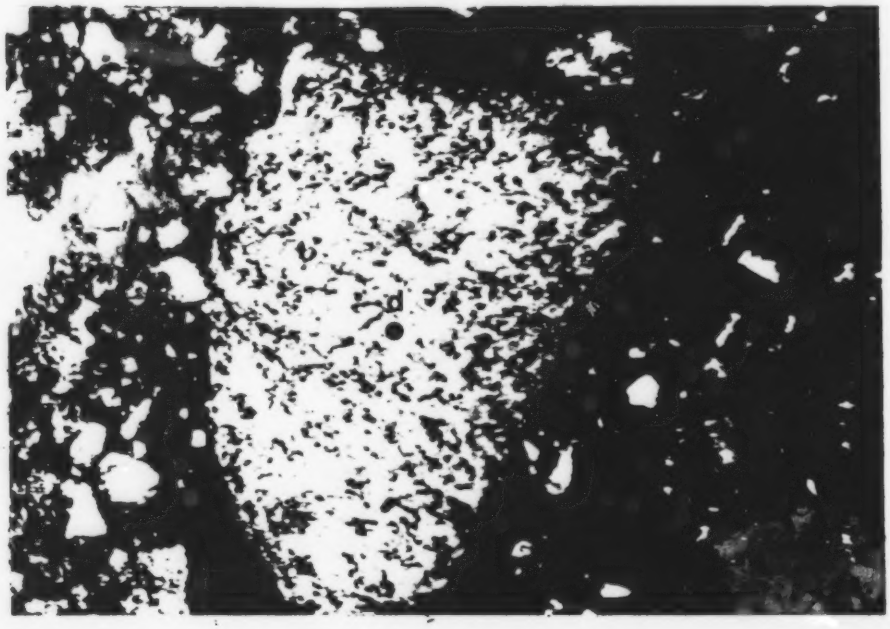


Fig. 1 - August 12/85 sample. Highly degraded vitrinite particle (d) Reflectance at (•) = .79. Polished surface, 1500 X, oil immersion.

Fig. 2 - August 12/85 sample. Degraded vitrinite particle (d). Reflectance at (•) = 1.00. Left: inertinite particle with reflectance at (•) = 1.60. Polished surface, 1500 X, oil immersion.

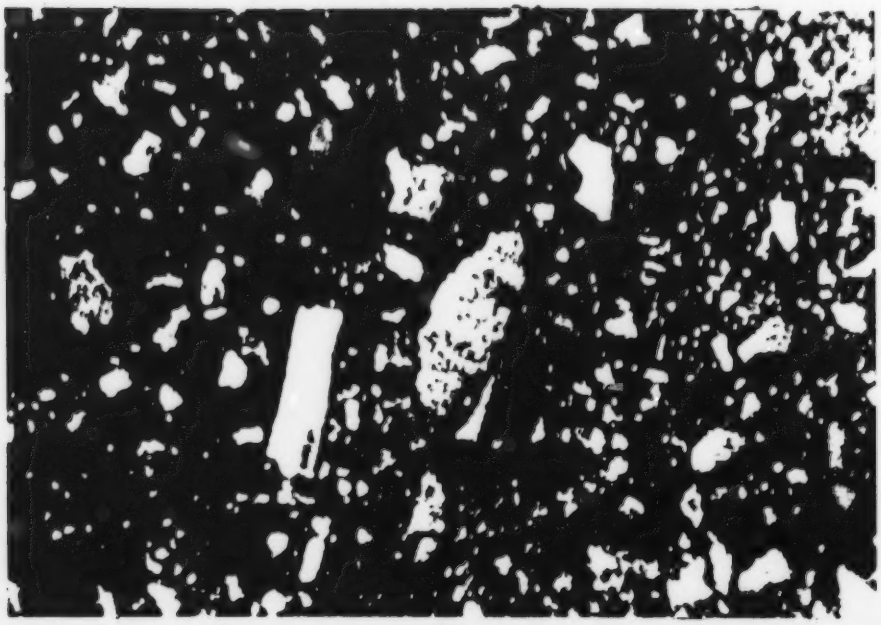


Fig. 3 - August 12/85 sample. The same field as Fig. 2 at lower magnification. Polished surface, 550 X, oil immersion.

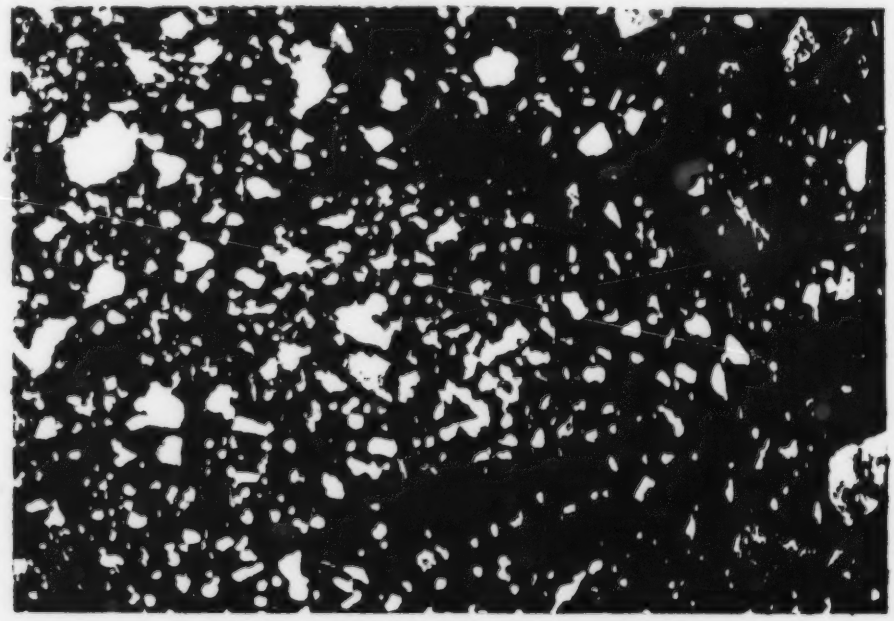


Fig. 4 - August 12/85 sample. Disintegrated coal particle. The subparticles remain together in close association with clays. Polished surface, 350 X, oil immersion.

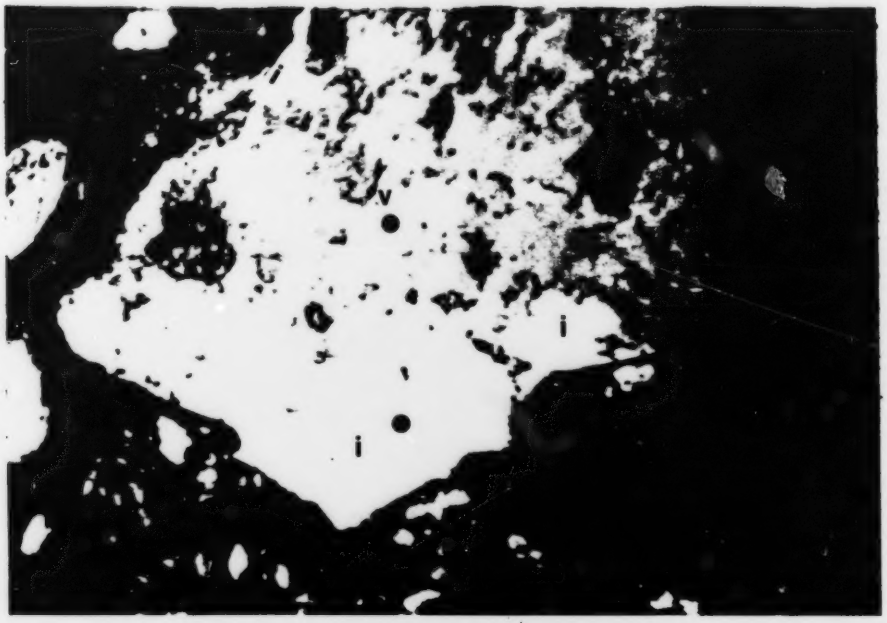


Fig. 5 - August 14/85 sample. Vitrinite (v) associated with inertinite (i). Reflectance of vitrinite at (•) = 1.05. Reflectance of inertinite at (•) = 1.64. Polished surface, 1500 X, oil immersion.

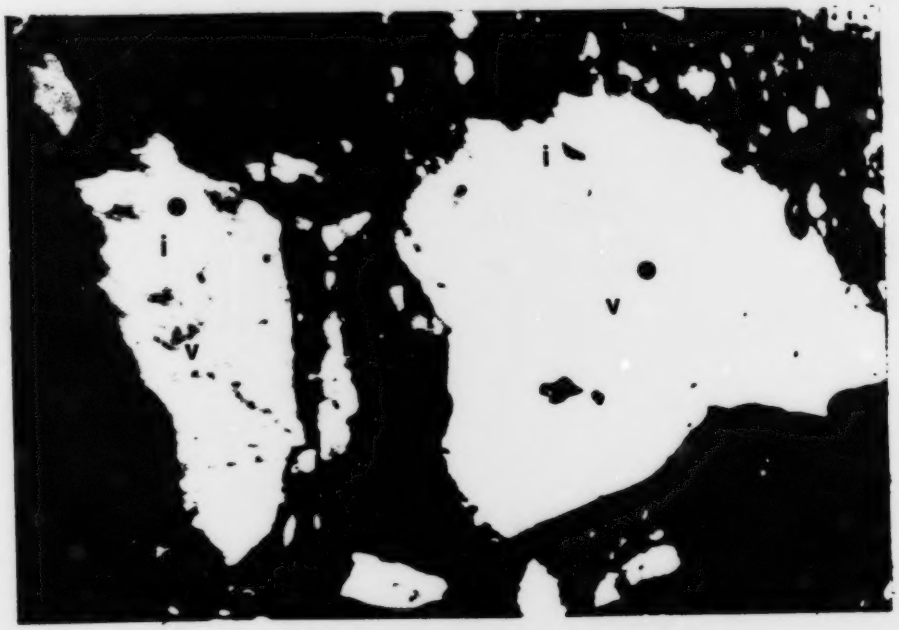


Fig. 6 - August 14/85 sample. Right: vitrinite (v) associated with inertinite (i). Reflectance of vitrinite at (•) 1.00. Left particle: reflectance of inertinite at (•) = 2.20. Polished surface, 1500 X, oil immersion.

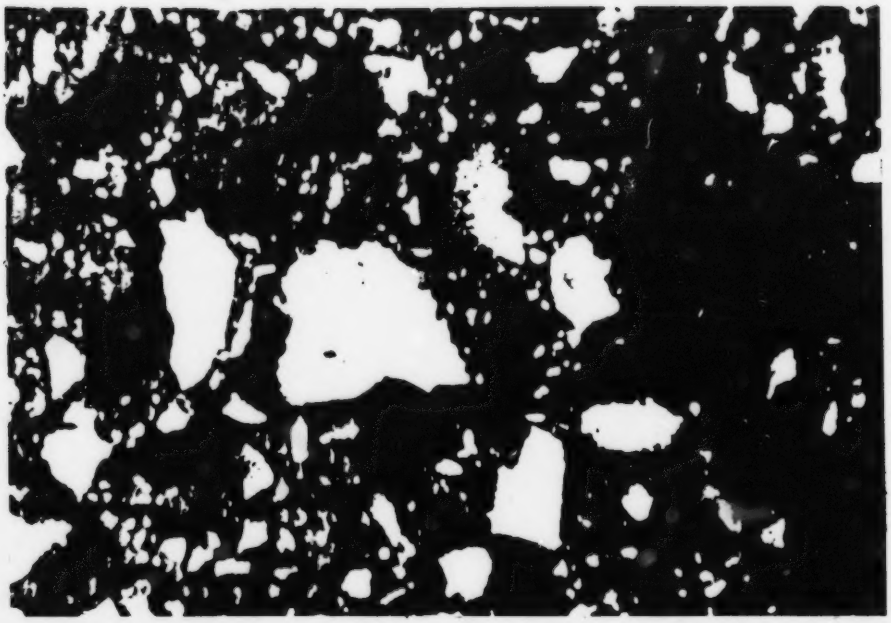


Fig. 7 - August 14/85 sample. The same field as Fig. 6 at lower magnification. Polished surface, 550 X, oil immersion.

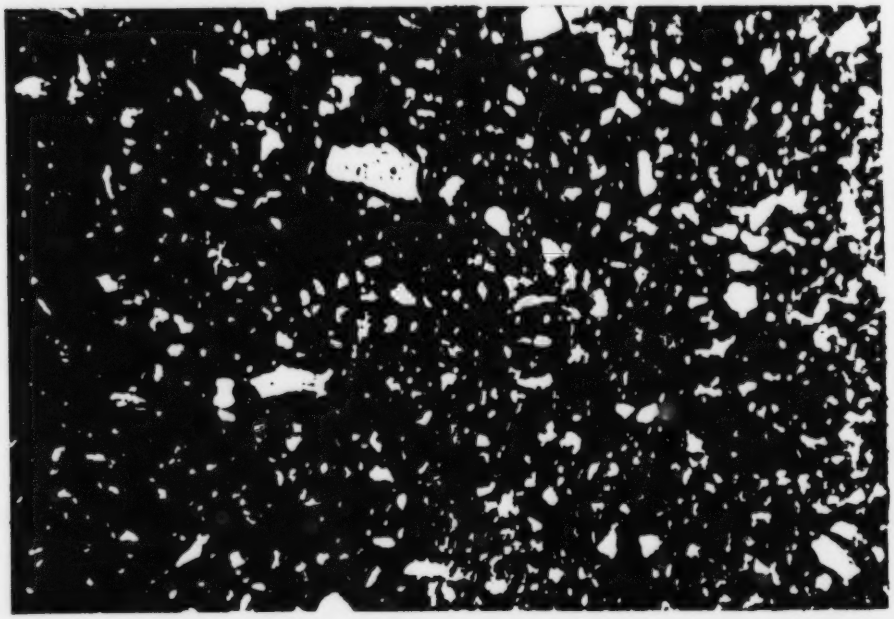


Fig. 8 - August 15/85 sample. General aspect of this sample. Polished surface, 550 X, oil immersion.

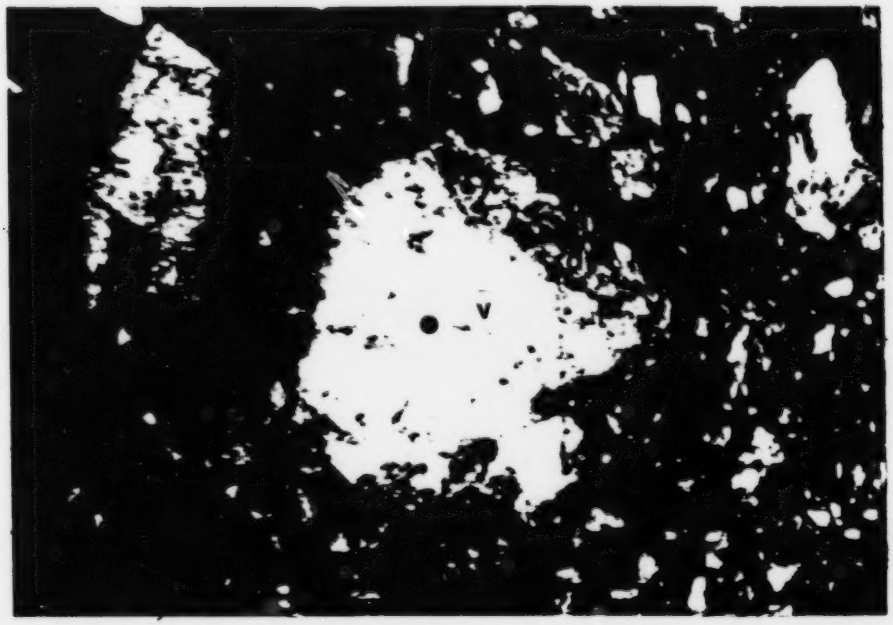


Fig: 9 - August 15/85 sample. Large vitrinite particle (v). Reflectance at (•) = 1.10. Note degradation on the top side of the particle (d). Top left: another particle with advanced stage of degradation. Polished surface, 1500 X, oil immersion.

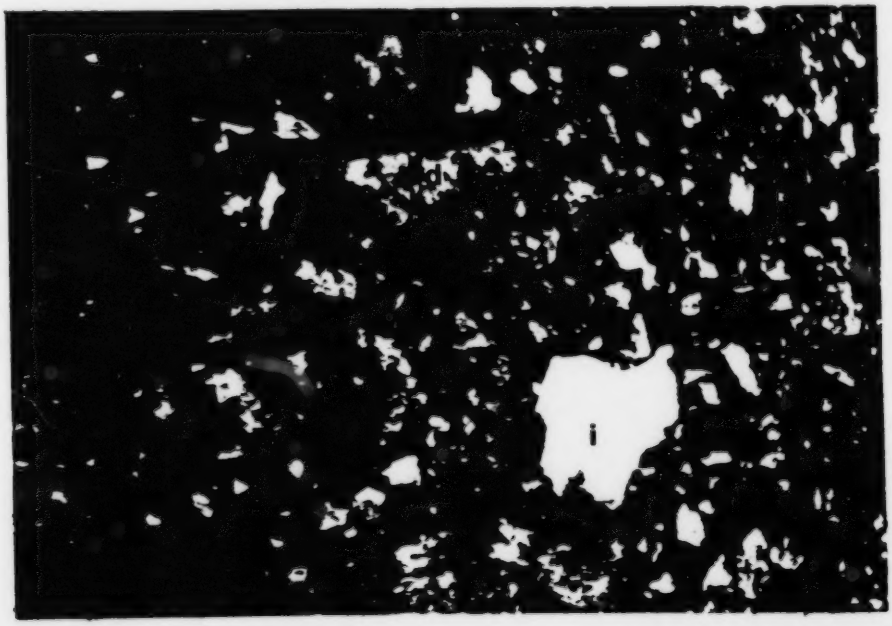
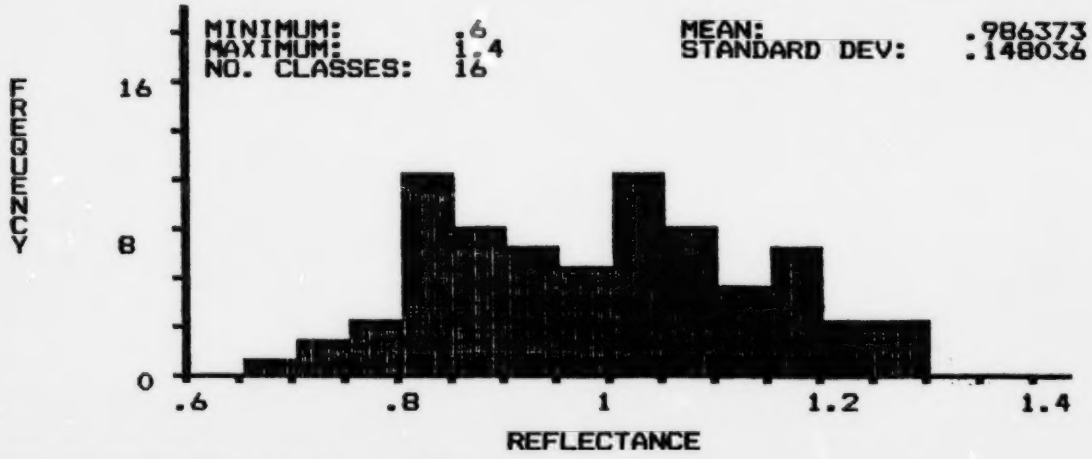


Fig 10 - August 15/85 sample. Inertinite particle (i) with reflectance 2.50. Top: highly degraded vitrinite particle (d). Polished surface, 1500 X, oil immersion.

Fig. 11 - Reflectogram of August 12 thickener feed sample



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CLASS LISTING

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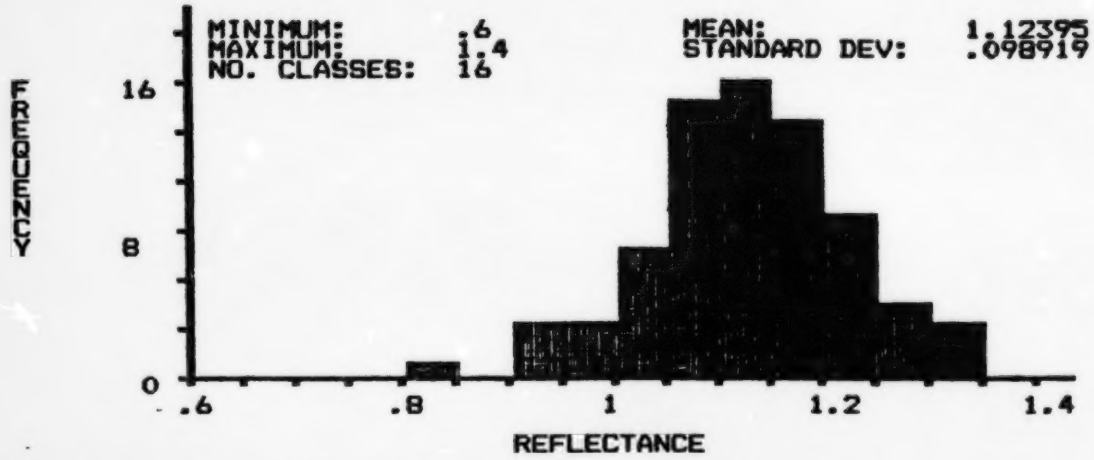
2	.65	.7	1	1.3
3	.7	.75	2	2.6
4	.75	.8	3	4
5	.8	.85	11	14.6
6	.85	.9	8	10.6
7	.9	.95	7	9.3
8	.95	1	6	8
9	1	1.04	11	14.6
10	1.04	1.08	8	10.6
11	1.08	1.13	5	6.6
12	1.13	1.17	7	9.3
13	1.17	1.21	3	4
14	1.21	1.26	3	4
15	1.26	1.31	0	0
16	1.31	1.35	0	0

Fig. 12 - Reflectogram of August 14 thickener feed sample



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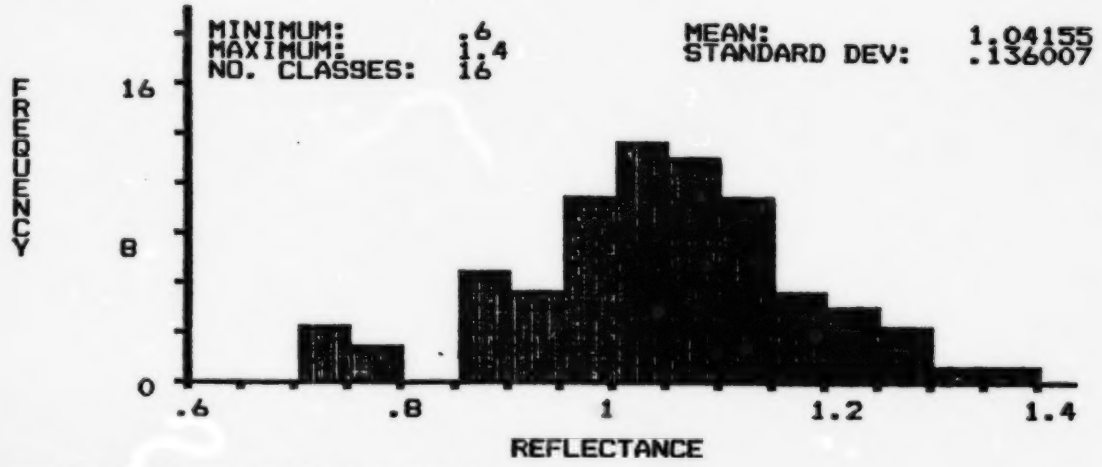
2	.65	.7	0	0
3	.7	.75	0	0
4	.75	.8	0	0
5	.8	.85	1	1.3
6	.85	.9	0	0
7	.9	.95	3	4
8	.95	1	3	4
9	1	1.04	7	9.3
10	1.04	1.08	15	20
11	1.08	1.13	16	21.3
12	1.13	1.17	14	18.6
13	1.17	1.21	9	12
14	1.21	1.26	4	5.3
15	1.26	1.31	3	4
16	1.31	1.35	0	0

Fig. 13 - Reflectogram of August 15 thickener feed sample



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6	.85	.9	5	6.6
7	.9	.95	10	13.3
8	.95	1	13	17.3
9	1	1.04	12	16
10	1.04	1.08	10	13.3
11	1.08	1.13	5	6.6
12	1.13	1.17	4	5.3
13	1.17	1.21	3	4
14	1.21	1.26	1	1.3
15	1.26	1.31	1	1.3
16	1.31	1.35		